PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



(51) International Patent Classification ⁶ :		(11) International Publication Number:	WO 99/42549
C11D 3/37, 11/02, 17/06	A 1	(43) International Publication Date:	26 August 1999 (26.08.99
 (21) International Application Number: PCT/US (22) International Filing Date: 19 February 1998 ((71) Applicant (for all designated States except US): THI TER & GAMBLE COMPANY [US/US]; One P Gamble Plaza, Cincinnati, OH 45202 (US). (72) Inventors; and (75) Inventors/Applicants (for US only): EBIHARA [JP/JP]; 6-3-8, Higashi-machi, Sakuragaoka, N Kobe 651-2225 (JP). MITO, Keiichi [JP/JP]; 1-/ Mikatadai, Nishi-ku, Kobe 651-2277 (JP). GOB/Tatsuo [JP/JP]; 4-11-3-604 Higashinakajima, H dogawa-ku, Osaka 533-0033 (JP). TAKEMORI [JP/JP]; 3-57, Kanda Minami Dori, Amagasaki (JP). (74) Agents: REED, T. David et al.; The Procter & Company, 5299 Spring Grove Avenue, Cincing 45217 (US). 	19.02.9 E PROProcter , Fuku Nishi-k 2-E-90 A YASF igashiy I, Shin 660-08	BY, CA, CH, CN, CU, CZ, DO GH, GM, GW, HU, ID, IL, IS LC, LK, LR, LS, LT, LU, LV MX, NO, NZ, PL, PT, RO, R TJ, TM, TR, TT, UA, UG, US patent (GH, GM, KE, LS, MW patent (AM, AZ, BY, KG, KZ, patent (AT, BE, CH, DE, DK, LU, MC, NL, PT, SE), OAPI CM, GA, GN, ML, MR, NE, S Published With international search report	E, DK, EE, ES, FI, GB, GB, JP, KE, KG, KP, KR, KZ, MD, MG, MK, MN, MW, U, SD, SE, SG, SI, SK, SI, UZ, VN, YU, ZW, ARIPP, SD, SZ, UG, ZW), Eurasia MD, RU, TJ, TM), Europea ES, FI, FR, GB, GR, IE, TI patent (BF, BJ, CF, CG, CSN, TD, TG).

ALATE

(57) Abstract

The present invention relates to an interspersion particle comprising, by weight of the interspersion particle, from about 20 % to about 50 % anionic surfactant and from about 15 % to about 30 % polymeric polycarboxylate; wherein the anionic surfactant and the polymeric polycarboxylate are interspersed within the interspersion particle; and wherein the weight ratio of the anionic surfactant to the polymeric polycarboxylate is from about 10:4 to about 10:9. Also disclosed herein are a particle composition and a cleaning composition comprising the interspersion particle and processes for making the interspersion particle.

THIS PAGE BLANK (USPTO)

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LÜ	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel .	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ.	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand		
CM	Cameroon		Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		•
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	Ц	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

WO 99/42549 PCT/US98/03280

AND A POLYMERIC POLYCARBOXYLATE

5

10

FIELD

The present invention relates to a particle having an anionic surfactant and a polymeric polycarboxylate.

15

25

BACKGROUND

There is a current trend for commercially available cleaning compositions, such as granular detergent compositions, to have higher bulk densities as well as higher active ingredient content. Such detergent compositions offer greater convenience to the consumer and at the same time, reduce the amount of packaging materials which will ultimately be disposed of. However, such cleaning compositions tend to encounter problems of poor dissolution. To address this problem, builder, filler and/or surfactant which have good water-solubility have been used in such high bulk density detergent. Unfortunately, although these materials help improve overall dissolution of composition, they also readily absorb moisture. Such affinity for moisture absorption tends to result in undesirable caking during manufacturing and storage of such compositions. Anionic surfactants, especially high level anionic surfactants, are a major facilitator of such caking. This caking is very undesirable for consumers.

10

15

20

Consequently, there is a need for high active materials used in cleaning compositions to have better physical properties, such as good dissolution, as well as low moisture absorption to minimize caking.

SUMMARY

The present invention relates to an interspersion particle comprising, by weight of the interspersion particle, from about 20% to about 50% anionic surfactant, and from about 15% to about 30% polymeric polycarboxylate, wherein the anionic surfactant and the polymeric polycarboxylate are interspersed within each individual particle, and wherein the weight ratio of the anionic surfactant to the polymeric polycarboxylate is from about 10:4 to about 10:9.

The invention also relates to compositions comprising one or more interspersion particles.

The invention also relates to a process for making the interspersion particle and/or compositions comprising one or more interspersion particles.

These and other features, aspects and advantages of the present invention will become evident to those skilled in the art from a reading of the present disclosure.

DETAILED DESCRIPTION

The interspersion particle of the present invention answers the need for high active materials for use in a cleaning composition, having better physical properties, such as good dissolution, as well as low moisture absorption to minimize caking. Without being bound by theory, it is believed potential caking of particles containing certain high active materials can be reduced or eliminated by interspersing polymeric polycarboxylate within individual particles containing the high active material. Such interspersion improves the overall physical property, such as anti-caking property, of such interspersion particles and compositions comprising the interspersion particles, including cleaning compositions as a whole which contain the interspersion particles. While the specification concludes with claims particularly pointing out and distinctly claiming the

15

20

25

30

invention, it is believed that the present invention will be better understood from the following description.

All ratios are weight ratios unless specifically stated otherwise.

As used herein, "comprising" means that other steps and other ingredients which do not affect the end result can be added. The terms encompass the terms "consisting of" and "consisting essentially of".

As used herein, "density" means bulk density unless specifically stated otherwise.

Herein, "interspersed" means the referenced components contained in the subject particle are substantially uniformly distributed throughout the particle.

Herein "cleaning composition" means any cleaning composition having the interspersion particle of the present invention. Cleaning composition includes a hand or machine laundry detergent composition including a laundry additive composition and a composition suitable for use in the soaking and/or pretreatment of stained fabrics; hard surface cleaning composition; and dish wash compositions.

Herein "particle/cleaning composition" means the interspersion particle of the present invention, the particle composition of the present invention, or the cleaning composition of the present invention.

All cited references are incorporated herein by reference in their entireties. Citation of any reference is not an admission regarding any determination as to its availability as prior art to the claimed invention.

The present invention relates to an interspersion particle comprising by weight of the interspersion particle, from about 20% to about 50% anionic surfactant and from about 15% to about 30% polymeric polycarboxylate. The anionic surfactant and the polymeric polycarboxylate are interspersed within each interspersion particle. The weight ratio of the anionic surfactant to the polymeric polycarboxylate is from about 10:4 to about 10:9.

The interspersion contains at a minimum, an anionic surfactant and a polymeric polycarboxylate interspersed within the interspersion particle. The

WO 99/42549 PCT/US98/03280

4

interspersion particle can optionally comprise other detersive ingredients. Such other detersive ingredients may make up the core of the interspersion particle, be interspersed within, and/or coat the interspersion particle. The interspersion particle preferably has a density of from about 200 to about 1200 grams per liter, and preferably from about 450 to about 950 grams per liter. The interspersion particle preferably has a mean particle size of from about 200 microns to about 800 microns.

One or more interspersion particles may form a particle composition.

A particle composition may comprises other detersive ingredients in addition to the interspersion particle, wherein the particle composition comprises at least about 50% by weight of the particle composition of the interspersion particles. In such particle compositions, such other detersive ingredients may be individual particles intermingled with the interspersion particles, and/or coat the interspersion particles and/or form an agglomerate with interspersion particles.

10

15

20

25

30

Other detersive ingredients include a cationic surfactant, a nonionic surfactant, an ampholytic surfactant, a zwitterionic surfactant, a builder, a bleaching agent, a softening agent, a dye transfer inhibitor, a soil release polymer, a brightener, a suds suppressor, an enzyme, and a perfume.

Other detersive ingredients in a particle composition also include an anionic surfactant and polymeric polycarboxylate, which can be the same as or different from that which is already interspersed in the interspersion particles.

In addition, interspersion particles and/or particle compositions may be included in a cleaning composition, preferably a detergent composition.

In one embodiment, a cleaning composition has at least one set of particles; preferably having at least a first set of particles and a second set of particles. At least one first set of particles is made up of interspersion particles comprising by weight of an individual interspersion particle from about 20% to about 50% anionic surfactant and from about 15% to about 30% polymeric polycarboxylate. The anionic surfactant and the polymeric polycarboxylate are interspersed within each interspersion particle. The weight ratio of the anionic

10

15

20

25

30

surfactant to the polymeric polycarboxylate is from about 10:4 to about 10:9. A second set of particles in such a cleaning composition may be selected from the group consisting of a second anionic surfactant, a cationic surfactant, a nonionic surfactant, an ampholytic surfactant, a zwitterionic surfactant, a second polymeric polycarboxylate, a builder, a bleaching agent, a softening agent, a dye transfer inhibitor, a soil release polymer, a brightener, a suds suppressor, an enzyme, a perfume and mixtures thereof. The first and second anionic surfactant may be the same or different, and the first and second polymeric polycarboxylate may be the same or different. The cleaning composition comprises at least about 50% by weight of the cleaning composition of the interspersion particles.

Anionic Surfactant

The interspersion particle contains an anionic surfactant. Nonlimiting examples of anionic surfactants useful herein include the conventional C₁₁-C₁₈ alkyl benzene sulfonates ("LAS") and primary ("AS"), branched-chain and random C₁₀-C₂₀ alkyl sulfates; the C₁₀-C₁₈ secondary (2,3) alkyl sulfates of the formula CH₃(CH₂)_x(CHOSO₃-M⁺) CH₃ and CH₃ (CH₂)_y(CHOSO₃-M⁺) CH_2CH_3 where x and (y + 1) are integers of at least 7, preferably at least 9, and M is a water-solubilizing cation, especially sodium; unsaturated sulfates such as oleyl sulfate; the C₁₂-C₁₈ alpha-sulfonated fatty acid esters; the C₁₀-C₁₈ sulfated polyglycosides; the C₁₀-C₁₈ alkyl alkoxy sulfates ("AE_xS"; especially EO 1-12 ethoxy sulfates); and the C₁₀-C₁₈ alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates). The C₁₂-C₁₈ betaines, sulfobetaines ("sultaines"), and C₁₀-C₁₈ amine oxides, can also be included in the overall compositions. C₁₀-C₂₀ conventional soaps may also be used. If high sudsing is desired, the branched-chain C₁₀-C₁₆ soaps may be used. Preferably, at least 40% of the anionic surfactant is a C11-C18 alkyl benzene sulfonates for good dissolution of interspersion particle.

The interspersion particle contains from about 20% to about 50%, preferably from about 30% to about 40%, by weight of the interspersion particle, anionic surfactant.

The particle composition or the cleaning composition may also include an anionic surfactant in addition to the anionic surfactant included in the interspersion particles.

Polymeric Polycarboxylates

5

10

15

20

25

The interspersion particle further includes a polymeric polycarboxylate. Such polymeric polycarboxylate useful herein include acrylic acid-based polymers and acrylic/maleic-based copolymers. Such acrylic acid-based polymers which are useful herein are the water-soluble salts of polymerized acrylic acid. The average molecular weight of such polymers in the acid form is preferably from about 3,000 to about 100,000, more preferably from about 6,000 to about 12,000. Water-soluble salts of such acrylic acid polymers can include, for example, the alkali metal, ammonium and substituted ammonium salts. Soluble polymers of this type are known materials. Use of polyacrylates of this type in cleaning compositions has been disclosed, for example, in Diehl, U.S. Patent 3,308,067, issued March 7, 1967.

Acrylic/maleic-based copolymers include the water-soluble salts of copolymers of acrylic acid and maleic acid. The average molecular weight of such copolymers in the acid form is preferably from about 3,000 to about 100,000, more preferably from about 5,000 to about 75,000, most preferably from about 6,000 to about 12,000. The ratio of acrylate to maleate segments in such copolymers will preferably range from about 30:1 to about 3:7, more preferably from about 7:3 to about 1:1. Water-soluble salts of such acrylic acid/maleic acid copolymers can include, for example, the alkali metal, ammonium and substituted ammonium salts. Soluble acrylate/maleate copolymers of this type are known materials which are described in European Patent Application No. 66915, published December 15, 1982, as well as in EP 193,360, published September 3, 1986, which also describes such polymers comprising hydroxypropylacrylate. Still other useful polymeric polycarbonate include the maleic/acrylic/vinyl alcohol terpolymers. Such materials are also

disclosed in EP 193,360, including, for example, the 45/45/10 terpolymer of acrylic/maleic/vinyl alcohol.

The interspersion particle contains from about 15% to about 30%, more preferably from about 17% to about 27%, by weight of the interspersion particle, a polymeric polycarboxylate. The weight ratio of the the anionic surfactant to the polymeric polycarboxylates is preferably from about 10:4 to about 10:9, more preferably from about 10:5 to about 10:8.

The particle composition or the cleaning composition may also include a polymeric polycarboxylate in addition to the polymeric polycarboxylate included in the interspersion particle.

Other Detersive Ingredients

10

15

20

25

30

The particle/cleaning composition of the present invention may optionally contain other detersive ingredients. The precise nature of these additional components, and levels of incorporation thereof will depend on the physical form of the composition, and the nature of the cleaning operation for which it is to be used.

Other Detersive Surfactants

Other detersive surfactants in addition to the above described anionic surfactant may be used. If a non-anionic (e.g., cationic, nonionic, ampholytic and/or zwitterionic) surfactant is used in a particle/cleaning composition, the particle/cleaning composition comprises preferably at least about 0.01% of a detersive surfactant; more preferably at least about 0.1%; further more preferably at least about 1%; more preferably still, from about 1% to about 50%, by weight of the particle/cleaning composition of other detersive surfactants.

Preferred other detersive surfactants are cationic, nonionic, ampholytic, zwitterionic, and mixtures thereof, further described herein below. Nonlimiting examples of detersive surfactants useful in the particle/cleaning composition include, the conventional nonionic and amphoteric surfactants such as the C₁₂-C₁₈ alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and C₆-C₁₂ alkyl phenol alkoxylates (especially ethoxylates and

10

15

25

mixed ethoxy/propoxy), C₁₂-C₁₈ betaines and sulfobetaines ("sultaines"), C₁₀-C₁₈ amine oxides, and the like, can also be included in the overall compositions. The C₁₀-C₁₈ N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the C₁₂-C₁₈ N-methylglucamides. See WO 9,206,154 to Cook, et al., published April 16,1992. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C₁₀-C₁₈ N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C₁₂-C₁₈ glucamides can be used for low sudsing. C₁₀-C₂₀ conventional soaps may also be used. If high sudsing is desired, the branched-chain C₁₀-C₁₆ soaps may be used. Mixtures of anionic and nonionic surfactants are especially useful.

Suitable cationic surfactants are preferably water dispersible compounds having surfactant properties comprising at least one ester (ie -COO-) linkage and at least one cationically charged group.

Other suitable cationic surfactants include the quaternary ammonium surfactants selected from mono C₆-C₁₆, preferably C₆-C₁₀ N-alkyl or alkenyl ammonium surfactants wherein the remaining N positions are substituted by methyl, hydroxyethyl or hydroxypropyl groups. Other suitable cationic ester surfactants, including choline ester surfactants, have for example been disclosed in US Patents No.s 4228042, 4239660 and 4260529.

20 Filler Salts

The particle/cleaning composition may also have filler salts. As one embodiment, the "compact" form of the particle/cleaning composition herein is best reflected by high density (e.g. 500g/liter to 950g/liter) and, in terms of granule, by a reduced amount of inorganic filler salt. Inorganic filler salts are conventional optional ingredients of detergent granules in powder form. In the particle/cleaning composition, the filler salt is preferably present in amounts not exceeding 25% of the particle cleaning composition, preferably not exceeding 15%, more preferably not exceeding 1% by weight of the particle/cleaning composition.

The inorganic filler salts are selected from the alkali and alkaline-earthmetal salts of sulfates and chlorides. A preferred filler salt is sodium sulfate.

Enzymes

5

10

15

20

25

30

The particle/cleaning composition may also have one or more enzymes which provide particle/cleaning performance and/or fabric care benefits.

Said enzymes include enzymes selected from, hemicellulases, peroxidases, proteases, gluco-amylases, cellulases, amylases, xylanases, lipases, esterases, cutinases, pectinases, reductases, oxidases, phenoloxidases, lipoxygenases, ligninases, pullulanases, tannases, pentosanases, malanases, ß-glucanases, arabinosidases chondroitinase, laccase or mixtures thereof.

Bleaching Agent

The particle/cleaning composition may also have bleaching agents such as anhydrous sodium perborate monohydrate, anhydrous sodium perborate tetrahydrate and percarbonate with a particle size of from about 400 to about 800 microns in diameter. These bleaching agent components can include one or more oxygen bleaching agents and, depending upon the bleaching agent chosen, one or more bleach activators. When present, oxygen bleaching compounds will preferably be present at levels of from about 1% to about 25% by weight of the particle/cleaning composition.

The bleaching agent component for use herein can be any of the bleaching agents useful for detergent compositions including oxygen bleaches as well as others known in the art. The bleaching agent suitable for the present invention can be an activated or non-activated bleaching agent.

One category of oxygen bleaching agent that can be used encompasses percarboxylic acid bleaching agents and salts thereof. Suitable examples of this class of agents include magnesium monoperoxyphthalate hexahydrate, the magnesium salt of meta-chloro perbenzoic acid, 4-nonylamino-4-oxoperoxybytyric acid and diperoxydodecanedioic acid. Such bleaching agents are disclosed in U.S. Patent 4,483,781 to Hartman, issued November 20, 1984, U.S. Patent Application 740,446 to Burns et al., filed June 3, 1985, European

15

20

25

30

Patent Application 0,133,354 to Banks et al., published February 20, 1985, and U.S. Patent 4,412,934 to Chung and Spadini, issued November 1, 1983. Highly preferred oxygen bleaches also include 6-nonylamino-6-oxoperoxycaproic acid (NAPAA) as described in U.S. Patent 4,634,551 to Hardy and Ingram, issued January 6, 1987.

The hydrogen peroxide releasing agents can be used in combination with bleach activators such as tetraacetylethylenediamine (TAED), nonanoyloxybenzene-sulfonate (NOBS, described in US 4,412,934 to Chung and Spadini, issued November 1, 1983), 3,5,-trimethylhexanoloxybenzenesulfonate (ISONOBS, described in EP 120,591) or pentaacetylglucose (PAG), which are perhydrolyzed to form a peracid as the active bleaching species, leading to improved bleaching effect. Also suitable activators are acylated citrate esters.

Bleaching agents other than oxygen bleaching agents are also known in the art and can be utilized herein. One type of non-oxygen bleaching agent of particular interest includes photoactivated bleaching agents such as the sulfonated zinc and/or aluminum phthalocyanines. These materials can be deposited upon the substrate during the washing process. Upon irradiation with light, in the presence of oxygen, such as by hanging clothes out to dry in the daylight, the sulfonated zinc phthalocyanine is activated and, consequently, the substrate is bleached. Preferred zinc phthalocyanine and a photoactivated bleaching process are described in U.S. Patent 4,033,718, issued July 5, 1977 to Holcombe, et al. Particle/cleaning compositions may contain about 0.025% to about 1.25%, by weight, of sulfonated zinc phthalocyanine.

Builder System

The particle/cleaning composition may also have a builder system.

Any conventional builder system is suitable for use herein including aluminosilicate materials, silicates and fatty acids, materials such as ethylenediamine tetraacetate, diethylene triamine pentamethyleneacetate, metal ion sequestrants such as aminopolyphosphonates, particularly ethylenediamine tetramethylene phosphonic acid and diethylene triamine

15

20

25

30

pentamethylenephosphonic acid. Though less preferred for obvious environmental reasons, phosphate builders can also be used herein where permitted.

Suitable builders can be an inorganic ion exchange material, commonly an inorganic hydrated aluminosilicate material, more particularly a hydrated synthetic zeolite such as hydrated zeolite A, X, B, HS or MAP. Another suitable inorganic builder material is layered silicate, e.g. SKS-6 (Hoechst). SKS-6 is a crystalline layered silicate consisting of sodium silicate (Na₂Si₂O₅).

Preferred builder systems for use in the particle/cleaning compositions include a mixture of a water-insoluble aluminosilicate builder such as zeolite A or of a layered silicate (SKS-6), and a water-soluble carboxylate chelating agent such as citric acid.

A suitable chelant for inclusion in the particle/cleaning compositions in accordance with the invention is ethylenediamine-N,N'-disuccinic acid (EDDS) or the alkali metal, alkaline earth metal, ammonium, or substituted ammonium salts thereof, or mixtures thereof. Preferred EDDS compounds are the free acid form and the sodium or magnesium salt thereof. Examples of such preferred sodium salts of EDDS include Na₂EDDS and Na₄EDDS. Examples of such preferred magnesium salts of EDDS include MgEDDS and Mg₂EDDS. The magnesium salts are the most preferred for inclusion in compositions in accordance with the invention.

Preferred builder systems include a mixture of a water-insoluble aluminosilicate builder such as zeolite A, and a water-soluble carboxylate chelating agent such as citric acid.

Other builder materials that can form part of the builder system for use in non-liquid compositions include inorganic materials such as alkali metal carbonates, bicarbonates, silicates, and organic materials such as the organic phosphonates, amino polyalkylene phosphonates and amino polycarboxylates.

Detergency builder systems are normally included in amounts of preferably from about 5% to about 60%, more preferably from about 10% to

about 50%, and further more preferably from about 20% to about 40% by weight of the particle/cleaning composition.

Softening Agents

5

10

15

20

25

30

The particle/cleaning composition may also have softening agents. These agents may be inorganic or organic in type. Inorganic softening agents are exemplified by the smectite clays disclosed in GB-A-1 400 898 and in U.S. Patent 5,019,292. Organic fabric softening agents include the water insoluble tertiary amines as disclosed in GB-A1 514 276 and EP-B0 011 340 and their combination with mono C₁₂-C₁₄ quaternary ammonium salts are disclosed in EP-B-0 026 527 and EP-B-0 026 528 and di-long-chain amides as disclosed in EP-B-0 242 919. Other useful organic ingredients of fabric softening systems include high molecular weight polyethylene oxide materials as disclosed in EP-A-0 299 575 and 0 313 146.

Levels of smectite clay are preferably in the range from about 2% to about 20%, more preferably from about 5% to about 15% by weight of the particle/cleaning composition, with the material being added as a dry mixed component to the remainder of the formulation.

Dve Transfer Inhibitors

The particle/cleaning composition may also have compounds, such as polymers, for inhibiting dye transfer from one fabric to another of solubilized and suspended dyes encountered during fabric laundering operations involving colored fabrics.

Especially suitable polymeric dye transfer inhibiting agents are polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, polyvinylpyrrolidone polymers, polyvinyloxazolidones and polyvinylimidazoles or mixtures thereof.

Other components may be employed, such as soil-suspending agents, soil-release agents, optical brighteners, abrasives, bactericides, tarnish inhibitors, coloring agents, suds suppressers, enzyme stabilizers, and/or encapsulated or non-encapsulated perfumes.

Process

The following describes four preferred types of processes for making the particle/cleaning composition of the present invention. The following examples further describe and demonstrate the preferred embodiments within the scope of the present invention.

In the examples, the abbreviated component identifications have the following meanings:

NaLAS	Sodium linear C ₁₂ alkyl benzene sulfonate
AS	Alkyl Sulfate
AE _X S	Alkyl Ethoxy Sulfate (X represents ethoxy number)
NaC _{X-Y} AS	Sodium C _X - C _Y alkyl sulfate
25EY	A C ₁₂₋ C ₁₅ predominantly linear primary alcohol
	condensed with an average of Y moles of ethylene oxide
NaSKS-6	Crystalline layered silicate of formula δ-Na ₂ Si ₂ O ₅
Phosphate or	Sodium tripolyphosphate
STPP	
MA/AA-A	MA/AA-A is known as "ML-8" from Nippon Shokubai KK
	(Japan), and is a copolymer of acrylic acid and maleic
	acid, having a molecular weight of 11,000, a mole ratio of
	acrylic to maleic of 60:40.
MA/AA-B	MA/AA-B is known as "KH4" from Nippon Shokubai KK,
	and is a copolymer of acrylic acid and maleic acid, having
	a molecular weight of 12,000, a mole ratio of acrylic to
1	maleic of 55:45.
MA/AA-C	MA/AA-C is known as "ML-7" from Nippon Shokubai KK,
	which is a copolymer of acrylic acid and maleic acid,
	having a molecular weight of 6,500, a mole ratio of acrylic
	to maleic of 70:30.

MA/AA-D	MAVAA-D is known as "Acusol 445N" from Rohm and
	Haas Company (Philadelphia, U.S.A.), which is a polymer
	of acrylic acid, having a molecular weight of 4,500.
MA/AA-E	MA/AA-E is a copolymer of acrylic acid and maleic acid,
	having a molecular weight of 60,000, a mole ratio of
	acrylic to maleic of 70:30.
NOBS	Nonanoyloxy benzene sulfonate in the form of the sodium
	salt
PB-4	Anhydrous sodium perborate tetrahydrate.
CMC	Sodium carboxymethyl cellulose
DTPA	Diethylene triamine penta acetate
PEG	Polyethylene glycol
PVP	Poly(vinylpyrrolidone)
Silicate	Amorphous Sodium Silicate (SiO ₂ :Na ₂ O ratio = 2.0)
Carbonate	Anhydrous sodium carbonate
Zeolite A	Hydrated Sodium Aluminosilicate of formula
	Na ₁₂ (A1O ₂ SiO ₂) ₁₂ . 27H ₂ O having a primary particle size
	in the range from 1 to 10 micrometers
Percarbonate	Anhydrous sodium percarbonate bleach of empirical
	formula 2Na ₂ CO ₃ .3H ₂ O ₂
Enzyme(s)	Enzyme(s) can be for example, pectinase, xylanase,
	protease, amylase, lipase, peroxidase and/or cellulase as
	follows.
Pectinase	Pectolytic enzyme sold under the tradename Pectinex AR
	by Novo Nordisk A/S.
Xylanase	Xylanolytic enzyme sold under the tradenames Pulpzyme
•	HB or SP431 by Novo Nordisk A/S or Lyxasan (Gist-
	Brocades) or Optipulp or Xylanase (Solvay).

Protease	Protease can be for example, Proteolytic enzyme sold
	under the tradename Savinase, Alcalase, Durazym by
	Novo Nordisk A/S, Maxacal, Maxapem sold by Gist-
	Brocades, Biosam sold by Showa Denko and proteases
	described in patents WO91/06637 and/or WO95/10591
	and/or EP 251 446, but is not limited to these examples.
Amylase	Amylolytic enzyme sold under the tradename Purafect Ox
	Am ^R described in WO 94/18314, WO96/05295 sold by
	Genencor; Termamyl®, Fungamyl®, Ban® and Duramyl®,
	all available from Novo Nordisk A/S and Maxamyl® by
	Gist-Brocades.
Lipase	Lipolytic enzyme sold under the tradename Lipolase,
	Lipolase Ultra by Novo Nordisk A/S
Peroxidase	Peroxidase enzyme
Cellulase	Cellulosic enzyme sold under the tradename Carezyme or
	Celluzyme by Novo Nordisk A/S.
SRP (Soil	Soil Release polymer can be, for example, sulfonated
Release	poly-ethoxy/propoxy end capped ester oligomer and
Polymer)	sulfobenzoyl and capped esters with oxyethyleneoxy and
	terephtaloyl backbone or methyl cellulose ether, but is not
	limited these examples.
Brightener	Bightener can be, for example, Disodium 4.4'-bis(4-anilino-
	6-morpholino-1,3.5-triazin-2-yl) amino) stilbene-2:2'-
	disulfonate, and disodium 4,4'-bis (2-sulphostyryl)
	biphenyl, but is not limited to these examples. Brightener
	can be a combination of two or more brighteners.
Suds	(1)mixture of 25% paraffin wax Mpt 50°C, 17%
Suppressor	hydrophobic silica and 58% paraffin oil;
	(2) mixture of 12% Silicone/silica, 18% stearyl alcohol and
·	70% starch in granular form; or
	(3) mixture of 5%silicone, 90% PEG and 5% water
Sulfate	Anhydrous sodium sulfate.

Example 1

10

15

20

25

30

This examples sets forth an exemplary process for making a composition having an interspersion particle. The process includes (A) making an aqueous slurry comprising an anionic surfactant and a polymeric polycarboxylate wherein the anionic surfactant and the polymeric polycarboxylate are dissolved in the aqueous slurry, and (B) spray-drying the aqueous slurry.

In step (A) above, the aqueous slurry is mixed uniformally thereby dissolving the anionic surfactant and the polymeric polycarboxylate in the aqueous slurry.

The aqueous slurry may further include carbonate, builder such as zeolite, cationic surfactant, sodium silicate and/or water. In step (B) above, a spray drying tower is preferably used for spray-drying. After the above spray-drying, compacting, grinding and/or coating steps may follow. The compacting is conducted by using a compacting machine (e.g., using Sinto compactor - BCS 25 type). The compacting includes (1) granulation and densification steps in a medium/high shear batch mixer/granulator, or (2) continuous granulation and densification steps (e.g., using Lodige® CB mixer and /or Lodige® KM mixer), (3) use of a fluid bed step, (4) use of a Compaction step (e.g., roll Compaction) and/or (5) use of an extrusion or kneader steps. Once formed, the medium to high density interspersion particle thus obtained by compacting and/or grinding step (5) can be coated by nonionic surfactant and/or builder or a flow aid such as zeolite A, and/or can be subsequently mixed with additives such as enzymes, bleach, perfume and crystalline layered silicate, etc. to obtain a particle composition and/or a cleaning composition.

Example 2

Alternatively, one could utilize a process which includes (A) making an aqueous slurry comprising an anionic surfactant wherein the anionic surfactant is dissolved in the aqueous slurry; (B) spray-drying the aqueous slurry to obtain a preliminary particle; and (C) compacting the preliminary particle and the polymeric polycarboxylate, wherein the polymeric polycarboxylate becomes

10

15

20

25

30

interspersed within the preliminary particle, thereby forming the interspersion particle.

In step (A) above, the aqueous detergent slurry may further include carbonate, builder such as zeolite A, cationic surfactant, sodium silicate and/or In step (A) above, the aqueous slurry is mixed uniformly thereby dissolving the anionic surfactant and the polymeric polycarboxylate in the aqueous slurry. In step (B) above, a spray drying tower is preferably used for spray-drying. In step (C) above, the compacting is conducted by using a compacting machine (e.g., using Sinto compactor - BCS 25 type). In step (C) above, the compacting intersperses the polymeric polycarboxylate within the interspersion particles. After the step (C) above, a grinding step may follow. The compacting includes (1) granulation and densification steps in a medium/high shear batch mixer/granulator, or (2) continuous granulation and densification steps (e.g. using Lodige® CB mixer and /or Lodige® KM mixer), (3) use of a fluid bed step, (4) use of a Compaction step (e.g., roll Compaction) and/or (5) use of an extrusion or kneader steps. Once formed, the medium to high density interspersion particle thus obtained by compacting can be coated by nonionic surfactant and/or builder or a flow aid such as zeolite A, and/or can be subsequently mixed with additives such as enzymes, bleach, perfume and crystalline layered silicate, etc. to obtain a particle composition and/or a cleaning composition.

Example 3

Alternatively, one could use a process which includes (A) mixing a polymeric polycarboxylate within a detergent agglomerate comprising a linear alkyl benzene sulfonic acid and an alkaline material during a (B) dryneutralization step, wherein a linear alkyl benzene sulfonic acid is neutralized in the presence of an alkaline material. Preferably, after the step above, a cooling step by a cooler may follow.

In mixing step (A), it is the use of a mixer under a dry-neutralization condition which intersperses the polymeric polycarboxylate within the

interspersion particle. The mixer useful herein can be, for example, a high speed mixer/densifier, or a variable-speed speed mixer/densifier. Alternatively, two or more mixers/densifiers can be used, for example, where a high speed mixer (e.g., a Lodige® CB mixer) is first used, and then a moderate speed mixer (e.g., Lodige® KM mixer) is used. The cooler useful herein can be, for example, a fluid bed cooler in which the detergent agglomerates are cooled and fines are removed. It is preferred that the detergent agglomerate has a density of from about 600 to about 950 grams per liter and a mean particle size of from about 250 microns to about 400 microns in diameter. It is preferred that the interspersion particle have a density of from about 550 to about 850 grams per liter and a mean particle size of from about 500 microns in diameter.

A non-liquid other surfactant can be further included with the builder. Preferred optional detersive ingredients include enzymes, brighteners, NOBS, PB-4, CMC, DTPA, perfume and soil-release polymer, and can be dry blended with the cooled interspersion particle to obtain a particle composition and/or a cleaning composition.

Example 4

10

15

20

25

Alternatively, one could use a process which includes (A) forming a wet detergent agglomerate by agglomerating a high active paste form of a surfactant with a polymeric polycarboxylate, an alkaline material, a builder, and other optional detersive ingredients in a high shear mixer followed by a medium shear mixer; (B) drying the wet detergent agglomerate to obtain a dried detergent agglomerate; and (C) further agglomerating the dried detergent agglomerate and a non-aqueous binder in a medium shear mixer, thereby forming the interspersion particles.

Specifically in the above step (A), a high active paste form of surfactant (e.g.70-80% active AS, AE_XS (X:1-12), NaLAS paste) is agglomerated with a polymeric polycarboxylate, sodium carbonate, builders (e.g. Zeolite A /STPP) and other inorganic and organic solids present in the formulation in a continuous

high shear mixer (e.g. Lodige® CB mixer) followed by further agglomeration in a medium shear mixer (e.g. Lodige® KM mixer). In step (B) above, the wet agglomerate is then preferably dried in a fluid bed drier. In step (C) above, the dried agglomerate is then mixed in a medium shear mixer (e.g. Lodige® KM mixer) and reagglomerated using a non-aqueous binder (e.g. PVP / PEG). Other detergent additives can be then mixed with the interspersion particle to obtain a particle composition and/or a cleaning composition.

Example 5

10

20

25

Alternatively, one could use a process which includes the following steps:

- (A) An aqueous slurry comprising anionic surfactants (NaLAS and Na C₁₄₋₁₅AS); cationic surfactant (coco-alkyl methyl bis (hydroxyethyl) ammonium chloride); polymeric polycarboxylate (MA/AA-A, MA/AA-B, MA/AA-C, MA/AA-D or MA/AA-E); Zeolite A (as builder); carbonate; silicate and/or sulfate is prepared by mixing in a crutcher. The slurry is then spray-dried in a spray-drier to obtain low density interspersion particles. The low density interspersion particles are then mixed in a mixer (e.g. KM mixer by Littleford, Inc.). The mixture is then compacted in a roll compactor. The roll compactor also increases the density of the mixture to form high density "chips." The high density chips from the compactor are then ground to the desired particle size distribution in a cage mill or a hammer mill to obtain a high density interspersion particle.
- (B) The high density interspersion particle is then coated with nonionic surfactants (e.g., 25E9). Then zeolite, precipitated silica (both are added as flow aids) and SRP are added to the interspersion particle coated by the nonionic surfactant. Perfume is then added thereto.
- (C) Other additives such as NaSKS-6, enzymes, brighteners, NOBS, percarbonate, and/or SRP are dry-added to these high density particle compositions and mixed to make the finished laundry detergent composition (cleaning composition). Compositions 1 through 7 are shown below and are made according to Example 5.

Step A	1	2	3	4	5	6	7
Composition							
(interspersion							
particle)							
NaLAS	28.53	29.00	29.00	25.00	15.00	28.53	28.53
NaC14-15AS	5.04	5.20	5.20	25.00	5.00	5.04	5.04
Sulfate	4.24	4.40	4.40	4.40	4.40	4.24	4.24
MM/AA-A	18.89	15.00	30.00	-	-	-	-
MM/AA-B	-	-	-	- ,	-	18.89	-
MM/AA-C	-	•	-	-	-	-	18.89
MM/AA-D	-	-	-	30.00	-	-	-
MM/AA-E	-	-	-	-	18.00	-	-
Cationic	1.58	1.60	1.60	1.60	1.60	1.58	1.58
surfactant							
Brightener(s)	0.37	0.30	0.30	0.30	0.30	0.37	0.37
Zeolite A (without	5.16	4.30	4.30	4.30	4.30	5.16	5.16
bound water)							
Silicate	16.29	17.00	17.00	5.00	17.00	16.29	16.29
Carbonate	14.71	20.00	5.00	1.20	31.20	14.71	14.71
Others	balance	balance	balance	balance	balance	balance	balance
•	100.00	100.00	100.00	100.00	100.00	100.00	100.00

(Each level of each ingredient in step A Composition is calculated as a solid.)

Step B Composition	1	2	3	4	5	6	7
(particle							
composition)							
Step A composition	87.75	87.00	87.00	87.00	87.00	87.75	87.75
25E9	3.78	3.80	3.80	3.80	3.80	3.78	3.78
Zeolite A (with bound	7.78	8.70	8.70	8.70	8.70	7.78	7.78
water) and Silica							
SRP	0.40	0.30	0.30	0.30	0.30	0.40	0.40
Perfume	0.29	0.20	0.20	0.20	0.20	0.29	0.29
•	100.00	100.00	100.00	100.00	100.00	100.00	100.00
•							
Step C Composition	1	2	3	4	5	6	7
(cleaning							
composition							
(finished detergent							
composition))							
Step B composition	86.61	86.40	86.40	86.40	86.40	86.61	86.61
Brightener(s)	· -	0.30	0.30	0.30	0.30	-	-
Enzyme(s)	0.57	1.30	1.30	1.30	1.30	0.57	0.57
Percarbonate	3.07	3.00	3.00	3.00	3.00	3.07	3.07
NaSKS-6	5.85	5.00	5.00	5.00	5.00	5.85	5.85
Suds Suppressor(s)	0.85	0.90	0.90	0.90	0.90	0.85	0.85
NOBS	2.70	2.70	2.70	2.70	2.70	2.70	2.70
SRP	0.35	0.40	0.40	0.40	0.40	0.35	0.35
•	100.00	100.00	100.00	100.00	100.00	100.00	100.00

The embodiments disclosed and represented by the previous examples have many advantages. For example, they can provide improved non-caking properties and minimal moisture absorption of a laundry detergent composition, they can reduce load of a grinder and a mixer due to less stickness of the particle

composition, and they can increase production capacity of a laundry detergent composition due to recycle rate reduction of a grinder and a mixer.

It is understood that the examples and embodiments described herein are for illustrative purposes only and that various modifications or changes in light thereof will be suggested to one skilled in the art and are to be included in the spirit and purview of this application and scope of the appended claims.

WHAT IS CLAIMED IS:

- 1. An interspersion particle comprising, by weight of the interspersion particle:
 - a. from about 20% to about 50% anionic surfactant, and
 - b. from about 15% to about 30% polymeric polycarboxylate;
- wherein the anionic surfactant and the polymeric polycarboxylate are interspersed within the interspersion particle, and wherein the weight ratio of the anionic surfactant to the polymeric polycarboxylate is from about 10:4 to about 10:9.
 - 2. A particle composition comprising one or more individual particles wherein at least one individual particle is an interspersion particle comprising, by weight of the interspersion particle:
 - a. from about 20% to about 50% anionic surfactant, and
- b. from about 15% to about 30% polymeric polycarboxylate;
 wherein the anionic surfactant and the polymeric polycarboxylate are interspersed within each interspersion particle, wherein the weight ratio of the anionic surfactant to the polymeric polycarboxylate is from about 10:4 to about 10:9; and wherein the particle composition comprises at least about 50% by weight of the particle composition of the interspersion particles.
 - 3. The particle composition of Claim 2, wherein at least 40% of the anionic surfactant is a C₁₁-C₁₈ alkyl benzene sulfonate.
 - 4. The particle composition of Claim 2, wherein the polymeric polycarboxylate is an acrylic/maleic-based copolymer having an average molecular weight of from about 3,000 to about 100,000 and a mole ratio of acrylic to maleic of from about 30:1 to about 3:7.

PCT/US98/03280 WO 99/42549

24

5

5. The particle composition of Claim 4, wherein the polymeric polycarboxylate is an acrylic/maleic-based copolymer having an average molecular weight of from about 6,000 to about 12,000 and a mole ratio of acrylic to maleic of from about 7:3 to about 1:1.

5

5

6. The particle composition of Claim 5, wherein acrylic/maleic-based copolymer is selected from the group consisting of a copolymer of acrylic acid and maleic acid having a molecular weight of about 11,000 and a mole ratio of acrylic to maleic of about 60:40; a copolymer of acrylic acid and maleic acid having a molecular weight of about 12,000 and a mole ratio of acrylic to maleic of about 55:45; a copolymer of acrylic acid and maleic acid having a molecular weight of about 6,500 and a mole ratio of acrylic to maleic of about 70:30; a copolymer of acrylic acid and maleic acid having a molecular weight of about 60,000 and a mole ratio of acrylic to maleic of about 70:30; and mixtures thereof.

10

7. The particle composition of Claim 2, wherein the polymeric polycarboxylate is a polymer of acrylic acid, having a molecular weight of about 4,500.

8.

The particle composition of Claim 2, wherein the interspersion particles have by weight of the interspersion particle from about 17% to about 27% polymeric polycarboxylate.

9.

A cleaning composition comprising the particle composition of any of Claims 2-8, further comprising other detersive ingredients wherein the cleaning composition comprises at least 50% by weight of the cleaning

composition of the interspersion particles.

- A process for preparing the particle composition of any one of Claims 2-8 10. comprising making an aqueous slurry having an anionic surfactant and a polymeric polycarboxylate wherein the anionic surfactant and the polymeric polycarboxylate are dissolved in the aqueous slurry, and spray-
- drying the aqueous slurry. 5

INTERNATIONAL SEARCH REPORT

Inter mal Application No PCT/US 98/03280

		<u>-</u>	
A. CLASS IPC 6	FICATION OF SUBJECT MATTER C11D3/37 C11D11/02 C11D17/	06	-
According to	o International Patent Classification (IPC) or to both national classific	ation and IPC	
	SEARCHED		·- ·- ·- ·- ·- ·- ·- ·- ·- ·- ·- ·- ·- ·
Minimum do IPC 6	ccumentation searched (classification system followed by classification ${\tt C11D}$	on symbols)	
	tion searched other than minimum documentation to the extent that s		
Electionic	ata base consulted during the international search (name of data ba	se and, where practical, search terms used)	
C. DOCUM	ENTS CONSIDERED TO BE RELEVANT		
Category '	Citation of document, with indication, where appropriate, of the rele	evant passages	Relevant to claim No.
X	EP 0 221 776 A (UNILEVER) 13 May see page 2, line 10 - line 31 see page 4, line 28 - line 56 see page 4, line 61 - line 63 see page 5, line 41 - line 44	1987	1-4,7-10 5,6
v	see page 6, line 16 - line 41 see page 20, line 19 - line 21 see page 23; table see claims 1,2		,
Y	WO 95 33815 A (PROCTER & GAMBLE) 14 December 1995 see page 3, line 5 see page 13, line 1 - line 4 see page 21; table see page 23, line 8 - line 11		5,6
Furth	er documents are listed in the continuation of box C.	X Patent family members are listed in	n annex.
° Special cat	egories of cited documents :	"T" later document nublished after the inter-	national filing date
conside	nt defining the general state of the art which is not ared to be of particular relevance ocument but published on or after the international	Tater document published after the inten or priority date and not in conflict with it cited to understand the principle or the invention	the application but ory underlying the
filing da	ate nt which may throw doubts on priority claim(s) or	"X" document of particular relevance; the cl cannot be considered novel or cannot involve an inventive step when the doc	be considered to
which i citation	S Cited to establish the publication date of another	"Y" document of particular relevance; the cl cannot be considered to involve an inv	almed invention entive step when the
other n "P" docume	neans nt published prior to the international filing date but	document is combined with one or more ments, such combination being obviou in the art.	s to a person skilled
	an the priority date claimed including search	"&" document member of the same patent f Date of mailing of the international sear	<u> </u>
15	o October 1998	23/10/1998	
Name and m	ailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,	Authorized officer	
	Fax: (+31-70) 340-3016	Magrizos, S	

INTERNATIONAL SEARCH REPORT

Information on patent family members

Inte. onal Application No PCT/US 98/03280

Patent document Publication cited in search report date		1	Patent family member(s)	Publication date	
EP 221776	Α	13-05-1987	AU AU	594091 B 6446986 A	01-03-1990 07-05-1987
		•	BR	8605393 A	11-08-1987
			CA	1297376 A	17-03-1992
			IN	166050 A	03-03-1990
			JP	6049879 B	29-06-1994
			JP	62112697 A	23-05-1987
			US	4900466 A	13-02-1990
WO 9533815	Α	14-12-1995	 AU ·	2035695 A	14-12-1995
			CA	2191564 A	14-12-1995
			EP	0763092 A	19-03-1997
			JP	10501283 T	03-02-1998
			US	5773401 A	30-06-1998